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IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Application No. : 10/712,257 Confirmation No. : 8056
First Named Inventor : Lothar ZIPFEL
Filed : November 14, 2003
TC/A.U. : 1711
Examiner : J. M. COONEY

Docket No. : 037110.52682US
Customer No. : 23911

Title : Non-Combustible Polyesterpolyol And/or Polyetherpolyol
Preblend for Producing Foamed Products

DECLARATION UNDER 37 C.F.R. § 1.132

Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

Sir:

I, Dr. Lothar Zipfel, hereby declare as follows:

1. I am a citizen of Germany, residing at 80880 Laatzen, Ilseeder Weg 11, Germany.
2. From 1972 to 1978, I studied chemistry at the University of Hannover, and I received my doctorate in 1981.
3. Starting on September 1, 1982, I was employed at what was then Kali-Chemie, and I have been the manager of Foam Application Technology at Solvay Fluor GmbH in Hannover since 1990.
4. I am familiar with the invention described in Claims 1-6 of the above-captioned patent application, which relates to a non-combustible premix for the production of foamed products.

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5. In our laboratories, we have demonstrated that the combination of a binary blowing agent (propellant mixture) and a phosphorus compound surprisingly increases the flashpoint of a polyol-based mixture resulting in a non-combustible product that is safe to transport and safe to handle.

6. The laboratory tests that are described herein were performed under my direction and supervision. According to our research, by combining commercially-available polyols with precisely 10-20 wt.% of a phosphorus compound and 4-35 wt.% of a binary blowing agent, a non-combustible premix is surprisingly formed. The non-combustibility can be demonstrated by measuring the flash point of the premix.

7. The flash points of the premixes described herein were measured using a Herzog HFP864 flame point apparatus, and the parameters for this testing were defined by the standard ISO 13736.

8. A binary blowing agent mixture comprising 93 wt.% 1,1,1,3,3-pentafluorobutane (hereinafter abbreviated "PFB") and 7 wt.% 1,1,1,2,3,3,3-heptafluoropropane (hereinafter abbreviated "HFP") was determined to be soluble in commercially available polyols such as Fox-o-pol VD280S, Fox-o-pol N470, Daltolac R530, PEC 400 and PPG 400.

9. An additional blowing agent comprising 1,1,1,3,3-pentafluoropropane (herein after abbreviated "PFP") can be added to the binary blowing agent

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described above to form a blowing agent mixture that also is soluble in commercially available polyols.

10. Table 1 shows the compositions of inventive pre-mixtures comprising a spray polyol, a catalyst, water, a phosphorus compound, and a binary blowing agent, wherein the phosphorus compound tris-chloroisopropyl phosphate is abbreviated "TCPP" and the phosphorus compound triethyl phosphate is abbreviated "TEP." The amount of each constituent in Table 1 is listed in grams.

11. In the three premix compositions shown in Table 1, the phosphorus compound comprises 11.7, 11.8 and 11.7 wt.%, respectively, and the binary blowing agent comprises 22.2, 21.9 and 22.2 wt.%, respectively, of the total mixture.

12. For Premixes 1 and 2, the expected flash point was between -10 and 0°C, and for Premix 3, the expected flash point was -10°C. Surprisingly, Premixes 1-3 of Table 1 were non-combustible mixtures and no flash point was measured.

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Table 1. Inventive, Non-Combustible Premix Compositions

| <u>Constituent (grams)</u> | <u>Premix 1</u> | <u>Premix 2</u> | <u>Premix 3</u> |
|----------------------------|-----------------|-----------------|-----------------|
| Spray Polyol #1 | 101.7 | 101.7 | 101.7 |
| Catalyst Mix #1 | 8.35 | 8.35 | 8.35 |
| Water | 2.5 | 2.5 | 2.5 |
| TOPP | 20 | 20 | 0 |
| TOPP:TEP (50:50) | 0 | 0 | 20 |
| PFB:HFP (93:7) | 37.9 | 29.6 | 57.9 |
| PFP | 0 | 7.5 | 0 |
| Expected Flash Point (°C) | -10°C-0°C | -10°C-0°C | -10°C |
| Measured Flash Point | None | None | None |

13. In a further experiment, the polyol Tercarol®, which is a polyether polyol available from Dow Chemical Company, was treated with various amounts of propellant. The amount of propellant with respect to the amount of polyol (wt.%) is shown in the first column of Table 2. For each concentration of propellant, four separate samples were made and the flash point was measured for each sample.

14. The second and third columns of Table 2 show the flash points of comparative samples wherein the propellant is either PFB or a 93:7 wt.% mixture of PFB and HFP. No phosphorus compound was added to these comparative samples. The fourth and fifth columns of Table 2 show the flash

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points of the liquid polyol mixtures of columns two and three further comprising 10 wt.% of the phosphorus compound TCPP.

15. As shown by the data in Table 2, the flash points of the various mixtures in columns 2 and 3 (no phosphorus compound) decreases with increasing propellant concentration. This is true for both the samples comprising PFB as well as the samples comprising the binary blowing agent mixture of PFB and HFP.

Table 2. Effect of Propellant and Phosphorus Compound on the Flash Point of Liquid Polyol Mixtures

| Propellant/Polyol(%) | PFB | PFB:HFP (93:7) | PFB+TCPP | PFB:HFP (93:7)+TCPP |
|----------------------|------------------|-------------------|----------|------------------------|
| Concentration (%) | Temperature (°C) | | | |
| 2.1 | 53.0 | 58.5 | 58.5 | 56.0 |
| 4.2 | 35.5 | 38.5 | 36.5 | 57.5 |
| 6.6 | 24.5 | 27.0 | 28.5 | 33.5 |
| 8.7 | 20.0 | 20.0 | 14.5 | 44.0 |
| 11.1 | 13.5 | 14.5 | 14.5 | -- |
| 17.7 | 1.5 | -- | 3.5 | -- |
| 25.0 | -3.5 | -- | -4.0 | No flash point |

16. As shown in column 4 of Table 2, the addition of TCPP to the mixtures comprising only the propellant PFB failed to markedly improve (increase) the flash points of these samples. By way of example, for the samples

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comprising 6.6%, 8.7% and 11.1% propellant, the flash point decreased 4.1%, decreased 27.5%, and only increased 7.4%, respectively, as a result of the addition of the phosphorus compound. A decrease in the flash point is an undesired result.

17. As shown in column 5 of Table 2, the addition of TCPP to the samples comprising the binary blowing agent mixture of PFB and HFP surprisingly and dramatically increased the flash points of these samples. We have discovered that the addition of specific proportions of a binary blowing agent (propellant mixture) and a phosphorus compound improves (increases) the flash point of the polyol-based mixture. The following examples from Table 2 are illustrative.

18. The addition of TCPP to the comparative sample comprising 2.1% binary blowing agent with respect to the polyol (corresponding to a binary blowing agent composition outside of the claimed range of 4-35%) resulted in a decrease in the flash point for this sample.

19. Surprisingly, the addition of TCPP to the samples comprising 6.6% and 8.7% binary blowing agent with respect to the polyol resulted in a substantial increase in the flash point for these samples (+24% and +120%, respectively). Significantly, an increase in the flash point was observed although the concentration of the binary blowing agent (propellant) was increasing.

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20. Typically, one would expect that if a critical amount of blowing agent is exceeded, a premix comprising the blowing agent would be readily combustible because of the low flash point of the mixture. For example, the samples comprising 25% HFC only (not a binary blowing agent) were extremely volatile either with or without the addition of the TCPP, and had flash points of -3.5 and -4°C, respectively. However, the addition of TCPP to the sample comprising 25% binary blowing agent with respect to the polyol surprisingly resulted in non-combustible sample.

21. The invention relates to a non-combustible premix. The cited references made of record in the Office Action do not teach or suggest that it is possible to obtain a non-combustible polyol-based premix, much less teach or suggest the critical proportions of a binary blowing agent (4-35 wt.%) and a phosphorus compound (10-20 wt.%) required to achieve such an unexpected result.

22. It would not have been obvious to one having ordinary skill in the art to prepare a non-combustible polyol-based premix having the claimed proportions of additives.

23. This Declaration is identical to the Declaration submitted on January 31, 2006, except for the correction of two clerical errors. The errors occurred in paragraph 7 and in Table 1. In paragraph 7, the model of the flame point apparatus has been changed from 384 to 364. In Table 1, the PFB:HFP

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ratio has been changed from (97:3) to (93:7). This change is consistent with the (93:7) PFB:HFP ratio disclosed in paragraph 14 and in Table 2.

24. All statements made herein of my own knowledge are true, and all statements made on information and belief are believed to be true, and further, these statements were made with the knowledge that willful statements and the like, so made, are punishable by fine or imprisonment, or both, under § 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the patent application or any patent issued thereon.

Respectfully Submitted,

Date: 14. Aug. 2006
Dr. Lothar Zipfel